Standard Operating Procedure for Total Hardness

LG502

Revision 01, March 2008

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Standard Operating Procedure for Dissolved Oxygen Micro Method, Winkler Titration

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.2 The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, use a sample aliquot containing not more than 25 mg $CaC0_3$.

1.3 Automated titration may be used.

2.0 SUMMARY OF METHOD

2.1 Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate (Na2EDTA). The end point of the reaction is detected by means of Eriochrome Black T indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.

3.0 BACKGROUND

3.1 Hardness of water is a measure of the total concentration of the calcium and magnesium ions expressed as calcium carbonate.

3.2 In this procedure, a water sample is buffered to pH 10.1 and indicator is then added to the buffered sample. The indicator, when added to a solution containing Ca and Mg ions turns red. EDTA, the titrant, complexes with Mg and Ca cations, removing them from association with the indicator. When all the Mg and Ca are complexed with EDTA, the indicator will turn blue.

3.3 The analysis must be performed on the mid depth sample during unstratified conditions, and on the mid-epilimnion and mid hypolimnion sample when stratification is present.

4.0 APPARATUS

4.1 Standard laboratory titrimetric equipment.

5.0 ANALYTICAL PROCEDURE

5.1 A 100 ml water sample is measured into a plastic beaker containing a stirring bar. The water should be at room temperature, so it is easiest to use the water warmed for specific conductance measurement.

5.2 A 1 ml volume of buffer solution is added to the stirred water. This buffer solution is found in the small bottle marked "Buffer Solution Hardness 1."

5.3 One packet of indicator "ManVer 2 Hardness Indicator Powder Pillows," is added to the buffered sample. A red color will result. **From this point, no more than 5 minutes should elapse to the end of the analysis** to prevent $CaCO_3$ formation.

5.4 While stirring, the sample is titrated with 0.01M EDTA solution until the sample turns blue(No tinge of red remains).

5.5 The total hardness is recorded on the bench sheet along with the date/time, and analyst.

5.6 The titrated sample, with a pH of approximately 10, is discarded into a holding container for future neutralization.

6.0 CALCULATIONS

6.1 Total Hardness; mg/L as CaCO3 = 10 x ml. of titrant.

7.0 QUALITY CONTROL

7.1 A control standard is analyzed at least once per lake. The control standard is prepared from the 1000 mg/L standard by diluting 50 ml in a 500 ml volumetric flask.

8.0 REFERENCES

8.1 These reagents and chemicals can be obtained from Hach Chemical Company and are described in Standard Methods for the examination of Water and Wastewater, 14th Edition.